

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
30 May 2003 (30.05.2003)

PCT

(10) International Publication Number  
**WO 03/043430 A1**

(51) International Patent Classification<sup>7</sup>: A23D 7/00, 7/015

(21) International Application Number: PCT/EP02/12180

(22) International Filing Date: 31 October 2002 (31.10.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
01204517.5 23 November 2001 (23.11.2001) EP

(71) Applicant (*for AL, AM, AT, AZ, BA, BG, BR, BY, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GE, HR, HU, ID, IS, JP, KG, KP, KR, KZ, LR, LT, LU, LV, MA, MD, MG, MK, MX, MZ, NO, PH, PL, PT, RO, RU, SE, SI, SK, TJ, TM, TN, TR, UA, UZ, VN, YU, BE, FR, GR, IT, MC, NL, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG only*): **UNILEVER N.V.** [NL/NL]; UNILEVER N.V., PATENT DEPARTMENT, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL).

(71) Applicant (*for AE, AG, AU, BB, BZ, CA, GB, GD, GH, GM, IL, KE, LC, LK, LS, MN, MW, NZ, OM, SD, SG, SL, TT, TZ, UG, VC, ZA, ZM, ZW, SZ, CY, IE only*): **UNILEVER PLC** [GB/GB]; UNILEVER HOUSE, Blackfriars, London, Greater London EC4 4BQ (GB).

(71) Applicant (*for IN only*): **HINDUSTAN LEVER LIMITED** [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Maharashtra, 400 020 Mumbai (IN).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **BOT, Arjen** [NL/NL]; Unilever R & D Vlaardingen, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). **FOSTER,**

**Timothy, John** [GB/GB]; Unilever R & D Colworth, Colworth House, Sharnbrook, Bedfordshire MK44 1LQ (GB). **LUNDIN, Leif, Orjan** [SE/GB]; Unilever R & D Colworth, Colworth House, Sharnbrook, Bath and South East Somerset MK44 1LQ (GB). **PELAN, Barbara, Margaretha, Catharina** [NL/NL]; Unilever R & D Vlaardingen, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). **REIFFERS, Christel** [NL/NL]; Unilever R & D Vlaardingen, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL).

(74) Agent: **VAN VELZEN, Maaïke, Mathilde**; Unilever NV, Patent Department, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL).

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: WATER CONTINUOUS ACIDIFIED FOOD PRODUCT

(57) Abstract: Acidified oil in water based food product which is a phase separated system comprising a biopolymer phase and a protein phase, wherein the volume fraction of the dispersed oil phase divided by the volume fraction of the protein phase is at least 0.25, preferably from 0.4 to 0.8, more preferred from 0.4 to 0.6.



WO 03/043430 A1

Water continuous acidified food product

### **Field of the invention**

The invention relates to a water continuous spreadable acidified food product suitable for use as a table spread which product comprises a fat phase consisting at least partly of vegetable oil or marine oil, biopolymer, protein and optionally further ingredients.

### **Background to the invention**

Water continuous spreads have been described in WO-A-97/04660 which discloses a creamy, cultured dairy based water continuous spread comprising less than 35% fat, up to 4.5% milk protein, gelatin or a gelatin replacer, the spread having a pH value between 4.6 and 5.2, and the spread having a butter-like mouthfeel, texture and taste.

Such spreads are popular for use as an underlayer on bread but are also consumed as such on toast and the like. These spreads in some aspects resemble well known fresh cheese and other dairy products.

Both the protein and the dairy fat contribute to the texture in these spreads, and it is possible to obtain products with the same firmness for different combinations of protein and fat concentrations. For dairy fat-based compositions, the high-protein / low-fat combinations are usually the most cost-effective solution to achieve maximal firmness. For compositions based on commonly used vegetable fat such as those disclosed in WO-A-97/08956, the situation is generally opposite: in those products the cost-effective solutions tend

2

to be the low-protein / high-fat formulations, because at current market prices dairy fat is much more expensive than most commonly used vegetable fats.

Improvement of firmness and texture of water continuous, oil containing spreads has been subject of many publications.

EP-A-864255 discloses very low fat spreads comprising a high amount of a fructo-oligosaccharide (from 1 to 20 wt%) leading to products where structure is given at least partly by this biopolymer. Such high levels however may influence the mouthfeel of these products negatively.

It is further for example well known that increase of fat content will lead to harder products (Jost et al., J. Food Sci. 51, 440, 1986; van Vliet, Coll. Polym. Sci. 266, 518, 1988; Langley and Green, J. Text. Studies. 20, 191, 1989; Xiong et al., J. Food Sci. 56, 920, 1991; Yost and Kinsella, J. Food Sci. 58, 158, 1993). The fat in these products plays a similar role as the 'filler phase' in a composite material. The effect of a fat droplet filler phase becomes more effective at higher filling fractions.

However the increase of the fat content is in many cases undesired as large groups of consumers nowadays prefer food products which are reduced fat compared to for example margarine but which still show the advantages of the high fat products. At these low filler fractions, however the contribution of the filler fraction to the firmness of the product is modest.

It is therefore an object of the current invention to provide a water continuous food product which contains a reduced amount

3

of fat, i.e. from 5 to 40 wt% fat, the right balance of protein and biopolymer to obtain a creamy mouthfeel, but for which the product firmness can be easily adjusted.

### **Summary of the invention**

It has surprisingly been found that those water continuous products that are based on a phase separated water phase comprising a biopolymer phase and a protein phase and that show a specific ratio between the volume fraction of the dispersed fat phase and the volume fraction of the protein phase, will meet at least part of the above objectives, especially in terms of firmness while using only a limited amount of fat.

Therefore the invention relates to a food product comprising a dispersed oil phase and a continuous aqueous phase said product comprising from 5 to 40 wt% fat, said fat being either a vegetable oil or marine oil or a combination thereof; or a combination of a dairy fat and a vegetable oil or marine oil, from 0.05 to 15 wt% protein, 0.01 to 3 wt% biopolymer, said food product having a pH value between 3.7, preferably 4.2 and 5.8, wherein the food product comprises a phase separated water phase comprising a biopolymer phase and a protein phase, wherein the volume fraction of the dispersed oil phase divided by the volume fraction of the protein phase is at least 0.2, preferably at least 0.25, more preferably at least 0.3.

In a further aspect the invention relates to a process for the preparation of these products.

### **Detailed description of the invention**

The invention relates to spreadable food products. Spreadable is defined as being easily spread with a knife on a substrate such as bread, without tearing the bread at the ambient temperature of the product during spreading. The products preferably are characterised by a Stevens hardness value hardness at 10 °C of about 40-700 g and of about 40-250 g at 20°C. The method to determine Stevens hardness is described in the examples. Preferred products show a Stevens hardness of from 50 to 500 g, more preferred 100 to 500 g at 5 °C and from 50 to 250 g at 20 °C.

In the description and claims where weight% is used this is weight% on total product weight unless otherwise is indicated.

In the description and claims the terms "oil" and "fat" are used interchangeably.

Volume fractions are defined on total product volume unless otherwise is indicated.

In the context of the invention protein phase is defined as the protein rich part of the water phase that has formed upon phase separation. In the context of the invention the products may comprise more than one protein enriched phase which can be separated due to physical barrier or may differ in type of protein. In the below the combination of protein phases is referred to as "the" protein phase.

In the context of the invention the biopolymer phase is defined as the protein depleted part of the water phase that has formed upon phase separation. Depending on the composition of the water phase more than one biopolymer phase may form. For the

purpose of the invention the combination of biopolymer phases is referred to as "the" biopolymer phase.

The invention relates to water continuous spreads containing a dispersed oil phase.

Firmness of these products is defined in terms of the so called Stevens hardness as mentioned above. The method to determine Stevens hardness is described in the examples.

It is well known that aqueous compositions comprising both proteins and biopolymers such as polysaccharides can present a phase separation. This means that above a certain concentration they no longer form a homogeneous mixture in aqueous medium but separate spontaneously in two phases; one phase enriched in biopolymers and one phase enriched in protein. The two phases can be quantified by centrifugation of a sample containing both ingredients in an aqueous medium.

Hence preferred products are those wherein the biopolymer and protein are thermodynamically incompatible compounds in an aqueous medium.

The products according to the invention comprise a phase separated water phase comprising a biopolymer phase and a protein phase. Without wishing to be bound by any theory it is believed that the protein is present in the form of an acidified protein network containing protein coated fat droplets which are the dispersed phase. The biopolymer phase is separately present and preferably forms the remainder of the aqueous phase.

The products according to the invention comprise a dispersed fat phase. Without wishing to be bound by any theory it is believed that in the products according to the invention, the



fat droplets are coated by protein and hence will mimic protein particles in many aspects. When studied under a microscope the products according to the invention preferably show a continuous aqueous phase in which a fat phase is dispersed in the form of fine droplets that are preferably at least partly coated with protein. Preferably at least 75% vol% , more preferred at least 90 vol% of the fat droplets is in the protein phase.

Optionally part of the fat droplets is located at the interface between the protein phase and the biopolymer phase.

Occasionally some fat droplets will be found in the biopolymer phase.

Most preferred essentially all of the fat droplets are within the protein phase.

Without wishing to be bound by any theory, it is believed that the phase separation leads to a concentration of the fat droplets in the protein phase. This concentration in turn enables a high influence of fat composition, especially in terms of solids, on the final product firmness.

Therefore the volume fraction of the dispersed oil phase divided by the volume fraction of the protein phase is at least 0.2, preferably at least 0.25, more preferably at least 0.3.

According to an even more preferred embodiment the volume fraction of the dispersed oil phase divided by the volume fraction of the protein phase is at least 1, more preferred from 1 to 2, most preferred from 1.2 to 2 when measured in the final product under acidic conditions (pH less than 6).

The average diameter  $D_{3,3}$  of the fat droplets is preferably from 0.1 to 20  $\mu\text{m}$ , more preferred from 0.5 to 5  $\mu\text{m}$  with sigma less

than 1, more preferred from 0.1 to 0.8. It is believed that the smaller the average diameter, the firmer the product will be.

The combination of a phase separated water phase with a dispersed fat phase of which the majority is present in only one of the two phases was found to lead to products for which the firmness is easily adjusted.

In the products according to the invention the biopolymer is present in the form of a biopolymer phase. Preferably the volume fraction of the biopolymer phase is from 0.2 to 0.5.

For the purpose of the invention the term biopolymer is defined such that it does not encompass protein. The biopolymer is selected from those biopolymers which phase separate with protein in an aqueous medium under the conditions of the current food product.

It will be appreciated that the selection of such biopolymer will depend on the protein that is applied. In general the following biopolymers tend to phase separate with protein in aqueous medium. Therefore the biopolymer is preferably selected from this group comprising locust bean gum , guar gum, tara gum, amylopectin, methylcellulose, alginate, starch, modified starch, high molecular weight pectin or combinations thereof.

Most preferably the biopolymer is selected from the group comprising locust bean gum , guar gum, tara gum, methylcellulose, alginate, or combinations thereof.

The concentration of biopolymer in food product according to the invention is from 0.01 to 3 wt%, preferably from 0.1 to 1.5 wt%. It will be appreciated that each individual biopolymer will have its own optimal concentration which may depend on



other characteristics of the food product such as the protein source, pH and salt content.

For example if locust bean gum is applied in combination with butter milk powder at a concentration of from 5 to 12 wt%, the concentration is preferably from 0.15 to 0.45 wt%.

The protein is preferably selected from the group of comprising milk protein, soy protein, pea protein or combinations thereof. The use of milk protein as at least part of the protein is highly preferred because of the positive effect of milk protein on the taste and flavour of the final product.

Suitable sources of milk protein are for example selected from the group comprising milk, skimmed milk powder, butter milk powder, butter serum powder, whey powder, whey powder, whey protein concentrate, whey protein isolate, caseinate. The most preferred protein is protein originating from butter milk because of its superb taste and flavour contribution.

The amount of protein is from 0.05 to 15 wt%, preferably from 2 to 10 wt%, more preferred from 2 to 6 wt%. In general the lowest possible protein concentration is most advantageous because of cost reasons.

The products according to the invention comprise from 5 to 40 wt% fat. Preferred products comprise 15 to 35 wt%, more preferred from 20 to 35 wt% fat.

The fat is either a vegetable oil or marine oil or a combination thereof; or a combination of a dairy fat and a vegetable oil or marine oil.

If dairy fat is used, the amount is preferably below 45% of the total fat. It has surprisingly been found that the firmness of the products can be adjusted accurately by adjusting the solids content of the fat. On the basis of generally known principles of the mechanical properties of composite materials, it was expected that the known measures of increase of fat content and protein content would influence the firmness of the final product. The unexpected large effect of solid fat content in the dispersed phase is surprisingly higher under the conditions of phase separation and phase volume of fat to protein in accordance with the current invention.

Preferably the solids content of the fat or fat blend that forms the dispersed phase is from 5 to 95% at 10 °C, from 1 to 50% at 20 °C and from 0 to 10% at 35 °C. More preferred the solids content is from 25 to 75% at 10 °C, from 7.5 to 35 at 20° C and from 0 to 5% at 35° C. Even more preferred the solids content is from 60 to 75% at 10 °C, from 10 to 35% at 20° C and from 0 to 5% at 35° C.

Even more preferred the same profile of solid fat is determined for the isolated fat phase of the product after it has been removed from the product. The method to determine solid fat content and the method to isolate the dispersed fat phase from the other ingredients of the product is disclosed in the example.

The above solid fat profile can be obtained by a variety of fats or combination of fats in a fat blend. The fat is preferably selected from the group comprising coconut oil, palm oil, palm kernel oil, soybean oil, rapeseed oil, sunflower oil, safflower oil, or fully or partially hardened fractions thereof.

More preferably the fat is selected from the group comprising coconut oil, hardened coconut oil, palm oil fractions or a combination thereof.

Optionally the fat is an interesterified fat blend. In a further preferred embodiment, the total amount of saturated fatty acid components in the fat is less than 45 wt%, based on the total amount of fatty acid components, and further preferred less than about 30 wt%.

Optionally the products according to the invention comprise emulsifier. For the purpose of the invention the term emulsifier does not encompass protein. However very high amount of emulsifier are preferably avoided as this could lead to a change in texture in terms of the contribution of the fat droplets to firmness of the product. Preferably the amount of emulsifier is below 1.3 wt%, more preferred below 1 wt%, even more preferred below 0.5%. Most preferred the product is essentially free of emulsifier. Suitable emulsifiers are for example monoglycerides (saturated or unsaturated), diglycerides, phospholipids such as lecithin, Tween<sup>tm</sup>, (sorbitan monostearate).

Optionally, usual additives for emulsions such as salt, herbs, spices, flavours, colouring matter, preservatives and the like may be added, although it is believed that for obtaining a suitable underlayer none of these is needed.

Normally, for use as a spread at least some salt will be present. The amount of salt may vary depending on the consumer preference in a specific country, but amounts between 0.2 and 1.5 wt% are generally recommended. The preferred salt is sodium chloride.

The products have a pH between about 3.7 and 5.8, preferably 4.2 to 5.8, more preferably between 4.5 and 5.2, and most preferred between 4.6 and 5.0.

Acidification of the starting ingredients to this pH can be obtained by any suitable method such as microbial acidification or chemical acidification for example using glucono deltalactone or another acidifying agent. The pH can be further adjusted by the use of a base such as sodium hydroxide.

For obtaining further improved spreadability and mouthfeel, in one embodiment of this invention preferably some gelatin will be present. The product preferably comprises at least 0.5 wt% gelatin (based on total weight of the product), and further preferred at least 0.6 wt%. No further beneficial effect was observed for levels above 2%, compared to 2% levels. It was found that if gelatin of a bloom strength of 250 is used, the best products are obtained if 0.8 - 1.2 wt% gelatin is used, based on fat free material. Preferred is to use 1.1 wt% gelatin. If gelatin of another bloom strength is used, other weight ranges are applied providing an equivalent structuring performance.

As these days it is sometimes desired to have no gelatin present in consumer products, a specific embodiment of this invention allows that instead of gelatin, a so called gelatin replacer is used. Gelatin replacers are components or compositions which have similar mouthfeel behaviour, and similar performance, such as water binding and melting properties compared with gelatin. Examples of suitable gelatin replacers are described in, inter alia, European Patent Application EP 496466 and in EP 474299 and are often very specific or specifically treated components or compositions.

The product according to the invention optionally comprises other ingredients such as herbs, flavour or colour components, gelatin.

It is also an object to provide a mildly, neutral tasting product having a closed keepability of several weeks. In a preferred embodiment, the products of the invention have a closed keepability of 8 weeks or more, which means that no change of taste and structure occurs on storage for such a period.

In a further aspect the invention relates to a process for the preparation of the above products. Any suitable process can be used provided that in at least one stage of the process phase separation between the protein phase and the biopolymer phase is obtained.

Therefore the invention also relates to a process for the preparation of a food product comprising a dispersed oil phase and a continuous aqueous phase said product comprising from 5 to 40 wt% fat, said fat being either a vegetable or marine fat or a combination thereof; or a combination of a dairy fat and a vegetable or marine fat, from 0.05 to 15 wt% protein in the form of a protein phase, 0.01 to 3 wt% biopolymer, having a pH value between about 3.7 and 5.8, preferably 4.2 and 5.8, said process comprising the steps of:

- a) preparation of an aqueous phase comprising protein and biopolymer
- b) mixing the aqueous phase with a fat phase at a temperature of about 40 to 70 °C
- c) heating the mixture obtained in step (b) for pasteurisation or sterilisation
- d) homogenisation of the mixture of step (c) at a pressure of

between 100 and 400 bar, preferably at a temperature above the melting point of the fat

e) acidification to a pH from about 3.7 to 5.8, preferably 4.2 to 5.8

f) homogenisation at a pressure of between 100 and 400 bar preferably at a temperature above the melting point of the fat.

Preferably during at least one stage of the process the biopolymer and protein phase separate .

The phase separation is preferably obtained by maintaining the pH in step (a) to (e) at from 5.2 to 8, preferably from 6.0 to 7.0. The optimal pH was found to be dependent among others on the isoelectric point of the protein. Phase separation is therefore preferably obtained at a pH above this point because at lower pH precipitation of the protein may result, especially at specific temperatures. An average isoelectric point is about 5.2. The pH may optionally be set higher than pH 8.

In case the products are acidified microbiologically it is preferred that the cultures are made inactive after the acidification. The product of the invention can contain some spore formers which are not destroyed by pasteurization, but cannot grow under the chilled storage conditions used for the presently claimed products.

Furthermore in case of microbiological acidification it is preferred that after step d) the composition is set to a temperature of from 5 to 50 °C.

After step (f) the products may be filled in containers either before or after including a cooling step to a temperature of from 5 to 10 °C.



For obtaining an increased closed keepability the product is filled into containers while at a temperature in excess of 65°C which containers then are hermetically sealed. By filling at a temperature in excess of 70°, a still better keepability is obtainable. By this higher temperature, the shelf life of the product in the closed container can be 8 weeks or even more.

In the process, acidifying and homogenization as indicated in step can be carried out in any order. It is preferred to homogenize at a temperature above 60°C.

The homogenisation in step (d) and (f) can be combined into one homogenisation step which is either carried out before or after acidification. The separation in two homogenisation steps is preferred.

According to another embodiment of the invention the food product is prepared in a process wherein at least part of and preferably all of the biopolymer is added after acidification.

In another aspect the invention relates to use of a fat which is at least partly crystallised at a temperature between 0 to 40 °C, to increase the firmness of an oil in water emulsion with 5 to 40 wt% fat.

According to the explanation provided above, it was surprisingly found that oil in water emulsions comprising a fat blend which is at least partly crystallised under the product's conditions, increases the firmness of the product, compared to a fat blend which is a liquid oil; i.e. which does not show crystallisation at any of the temperatures between 0 and 40 °C.

Also it was unexpectedly found that increasing the solid fat content of the dispersed fat phase in oil in water emulsions

15

comprising from 5 to 40 wt% increases the firmness of the products.

The fats that are at least partly crystallised at a temperature between 0 and 40 °C are preferably vegetable fats or comprise a combination of vegetable fat and dairy fat. Most preferred the solids content of the fat or fat blend that forms the dispersed phase is from 5 to 95% at 10 °C, from 1 to 50% at 20 °C and from 0 to 10% at 35 °C. More preferred the solids content is from 25 to 75% at 10 °C, from 7.5 to 35% at 20° C and from 0 to 5% at 35° C. Even more preferred the solids content is from 60 to 75% at 10 °C, from 10 to 35 at 20° C and from 0 to 5% at 35° C.

## Examples

### General

#### Method to determine $D_{3,3}$

The fat droplet size was measured using a well known low resolution NMR measurement method. Reference is made to Goudappel, G.J.W. et al; Journal of colloid and interface science 239, 535-542 (2001).

#### Method to determine solid fat content

The solid fat content (%) can be measured by a suitable analytical method such as NMR. The method used is low resolution NMR with Bruker Minispec apparatus. Reference is made to the Bruker minispec application notes 4, 5 and 6.

The percentage of solid fat determined by the low resolution NMR technique is defined as the ratio of the response obtained

from the hydrogen nuclei in the solid phase and the response arising from all the hydrogen nuclei in the sample. The product of this ratio and one hundred is termed the low resolution NMR solids percent. No correction is made for variations in the proton density between solid and liquid phase. The NMR solids percent for a sample measured at  $t$  °C was given the symbol  $N_t$ .

Suitable instruments adapted to determine the solids fat content are the Bruker Minispecs p20i<sup>tm</sup>, pc20<sup>tm</sup>, pc120<sup>tm</sup>, pc120s<sup>tm</sup>, NMS120<sup>tm</sup> and MQ20<sup>tm</sup>.

Stabilization and tempering procedure was as follows:

- melt fat at 80 °C
- 5 minutes at 60 °C
- about 1 day at 0 °C
- 30-35 minutes at each chosen measuring temperature.

#### Determination of phase separation

The preferred method is the method where phase separation is determined under acidic conditions in the final product. According to this method product was poured into tubs and centrifuged at about 1.000 to 5.000 g at 30 °C until phase separation was complete. The preferred force is around 3.000 g.

In an alternative embodiment the aqueous phase comprising biopolymer and protein, before acidification under neutral conditions, was poured in tubes that were centrifuged at 50°C for 2 h at a speed of 1053 rpm using a Gerber centrifuge.

For each method phase volumes for upper biopolymer-rich and lower protein-rich phase were quantified for each tube.

Protein analysis in biopolymer and protein phases

The protein content of the LBG and protein phases after centrifugation was analysed using the Kjeldahl method.

Stevens hardness

The firmness of the products is determined by measuring the force required to penetrate a cylindrical probe in the product. Sample height 5 cm; cylindrical probe of 0.5 inch thickness; compression rate 2 mm/s; penetration depth 20 mm. The samples are stored for 7 days at 5 °C, and stored at 5, 10, 20, 25, or 35 °C for 4 h before the firmness measurement.

Table 1 : Compositions

Ingredient	Wt % on product
Fat	25.0
Butter Milk Powder (BMP)	10.0
Locust Bean Gum (LBG)	0.3
Gelatin	0.7
Salt	0.3
Lactic acid (LA 88% pure)	0.58
Demineralised water	Up to 100%

The fat type varied for example 1-4

**Example 1:**

Fat blend: sunflower oil; N line:

18

Solids content at 10 °C (N10): 0

Solids content at 20 °C (N20): 0

Solids content at 35 °C (N35): 0

#### **Example 2**

Fat blend: mixture of sunflower oil, hardened coconut oil and a palm oil fraction; N line:

Solids content at 10 °C (N10): 25.6

Solids content at 20 °C (N20): 7

Solids content at 35 °C (N35): 0

#### **Example 3**

Fat blend: mixture of hardened coconut oil and a palm oil fraction; N line:

Solids content at 10 °C (N10): 64.9

Solids content at 20 °C (N20): 12.5

Solids content at 35 °C (N35): 0.3

#### **Example 4**

Fat blend according to example 2 but containing 15% BMP at constant LBG level.

#### Process

Water phase and fat phase ingredients except for acids were mixed at about 60 °C. After mixing the composition was pasteurized at 85°C for 10 minutes, and cooled down to 44°C, after which homogenisation at 200 bar took place. To the homogenized composition acid was added, until a pH of about 4.8 was reached. Followed by heating the mixture to 85 °C. The obtained product was homogenized at 300 bar, and subsequently heated to a temperature of 75°C for filling the small

19

containers. The product was cooled down to below 10°C and stored at chill temperature.

#### Results:

Parameter	Example 1	2	3	4
Stevens value at 5 °C	74.5	203.5	278.8	262.0
Phase volume oil phase (Po)	0.25	0.25	0.25	0.25
Phase volume protein phase (Pp)	About 0.14 <sup>a</sup> 0.375 <sup>b</sup>	About 0.14 <sup>a</sup> 0.375 <sup>b</sup>	About 0.14 <sup>a</sup> 0.375 <sup>b</sup>	About 0.21 <sup>a</sup> 0.44 <sup>b</sup>
Po divided by Pp <sup>c</sup>	About 1.8	About 1.8	About 1.8	About 1.2

- a: determined with preferred method under acidic conditions.
- B: determined under neutral conditions, before acidification
- C: determined using value (a).

It is clear from the above data that the increase of solids content of the fat blend in ex 1-3 leads to increased products firmness.



**Example 5**

A product was prepared according to the process of example 1-4.

Composition (wt%):

- 0.45% whey protein from 3.0% Sweet Whey Powder (powder contained 15% protein)
- 4.68% soy protein from 5.5% Soy Protein Isolate (powder contained 85% protein)
- 0.7% guar powder
- 26% fat

Fat blend was a blend of sunflower oil and an interesterified blend of palm oil and palm kernel oil:

T(°C)	Solid Fat Content(%)
5	25.7
10	22.9
15	18.5
20	14.2
25	10.6
30	7.9
35	4.9
40	2.0
45	0.0
50	0.0

The resulting product showed a Stevens firmness at 5°C: 186 +/- 15 g.

Phase volume distribution:

- protein: about 0.2 when determined under acidic conditions with preferred method and about 0.41 protein phase volume when determined under neutral conditions.
- thickener: 0.33
- fat: 0.26
- The ratio of phase volume of oil phase to protein phase was about 1.3.

**Example 6**

Composition:

Ingredient	Example 6	7	8
Fat (mixture of palm oil and coconut oil)	22	27	8
Milk protein (skim milk powder and whey protein isolate, whey protein to casein ratio is about 1)	3.43	5.13	6.6
Locust bean gum	0.3	0.24	0.3
salt	0.3	0.3	0.3
Potassium sorbate	0.1	0.1	0.1
acid	To pH 4.8	To pH 4.8	To pH 4.8
water	Up to 100wt%	Up to 100 wt%	Up to 100 wt%

Results:

Phase volume oil phase divided by protein phase:

Example 6: about 2

Example 7: about 1.8

Example 8: about 0.3

Stevens value at 5 °C:

Example 6: 156 g

Example 7: 609 g

Example 8: below 40 g

**Claims**

1. Food product comprising a dispersed oil phase and a continuous aqueous phase said product comprising from 5 to 40 wt% fat, said fat being either a vegetable oil or marine oil or a combination thereof; or a combination of a dairy fat and a vegetable oil or marine oil, from 0.05 to 15 wt% protein, 0.01 to 3 wt% biopolymer, said food product having a pH value between 3.7 and 5.8, preferably between 4.2 and 5.8, wherein the food product comprises a phase separated water phase comprising a biopolymer phase and a protein phase, wherein the volume fraction of the dispersed oil phase divided by the volume fraction of the protein phase is at least 0.2, preferably at least 0.25, more preferably at least 0.3.
2. Food product according to claim 1 wherein the biopolymer is present in the form of a biopolymer phase and wherein the volume fraction of the biopolymer phase is from 0.2 to 0.5.
3. Food product according to any of claims 1-2 wherein the volume fraction of the dispersed oil phase divided by the volume fraction of the protein phase is at least 1, more preferred from 1 to 2, most preferred from 1.2 to 2.
4. Food product according to any of claims 1-3 wherein the biopolymer and protein are thermodynamically incompatible compounds in an aqueous medium.
5. Food product according to any of claims 1-4 wherein the biopolymer is selected from the group comprising locust bean gum , guar gum, tara gum, amylopectin,

23

methylecellulose, alginate, starch, modified starch, high molecular weight pectin or combinations thereof.

6. Food product according to any of claims 1-5 wherein the protein is selected from the group comprising milk protein, soy protein, pea protein or combinations thereof.
7. Food product according to any of claims 1-6 wherein the amount of fat is from 15 to 35 wt%, more preferred from 20 to 35 wt%.
8. Food product according to any of claims 1-7 wherein the fat phase after isolation from the product is characterised by a solids content of from 60 to 75% at 10 °C, from 10 to 35% at 20° C and from 0 to 5% at 35° C.
9. Food product according to claim 8 wherein the fat is selected from the group comprising coconut oil, hardened coconut oil, palm oil fractions or a combination thereof.
10. Process for the preparation of a food product comprising a dispersed oil phase and a continuous aqueous phase said product comprising from 5 to 40 wt% fat, said fat being either a vegetable or marine fat or a combination thereof; or a combination of a dairy fat and a vegetable or marine fat, from 0.05 to 15 wt% protein in the form of a protein phase, 0.01 to 3 wt% biopolymer, having a pH value between 3.7 and 5.8, preferably 4.2 and 5.8, said process comprising the steps of:
  - a) preparation of an aqueous phase comprising protein and biopolymer
  - b) mixing the aqueous phase with a fat phase at a temperature of about 40 to 70 °C
  - c) heating the mixture obtained in step (b) for

pasteurisation or sterilisation

d) homogenisation of the mixture of step (c) at a pressure of between 100 and 400 bar, preferably at a temperature above the melting point of the fat

e) acidification to a pH between 3.7 and 5.8, preferably 4.2 to 5.8

f) homogenisation at a pressure of between 100 and 400 bar preferably at a temperature above the melting point of the fat.

11. Process according to claim 10 wherein during at least one stage of the process the biopolymer and protein phase separate.
12. Process according to claim 10 wherein phase separation is obtained by maintaining the pH in step (a) to (e) at from 5.2 to 8, preferably from 6.0 to 7.0.
13. Use of a fat which is at least partly crystallised at a temperature between 0 to 40 °C, to increase the firmness of an oil in water emulsion with 5 to 40 wt% fat.
14. Use according to claim 14 wherein the fat is a vegetable or the fat comprises a combination of vegetable fat and dairy fat.

## INTERNATIONAL SEARCH REPORT

Inte      nal Application No

PCT/EP 02/12180

A. CLASSIFICATION OF SUBJECT MATTER  
 IPC 7    A23D7/00    A23D7/015

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7    A23D    A23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, FSTA, BIOSIS

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 97 04660 A (UNILEVER PLC ;UNILEVER NV (NL)) 13 February 1997 (1997-02-13) cited in the application claims; example 3 ---	1-10
A	WO 97 08956 A (UNILEVER PLC ;UNILEVER NV (NL)) 13 March 1997 (1997-03-13) cited in the application claims ---	1-10
A	EP 0 864 255 A (ST IVEL LTD) 16 September 1998 (1998-09-16) cited in the application page 2, line 55 -page 3, line 10 ---	1-10
A	WO 96 03888 A (UNILEVER PLC ;UNILEVER NV (NL)) 15 February 1996 (1996-02-15) the whole document ---	1,5-10
	--- -/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

## \* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*Z\* document member of the same patent family

Date of the actual completion of the international search

14 February 2003

Date of mailing of the international search report

04/03/2003

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
 NL - 2280 HV Rijswijk  
 Tel (+31-70) 340-2040, Tx. 31 651 epo nl,  
 Fax: (+31-70) 340-3016

Authorized officer

Grittern, A



## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 02/12180

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 39 07 676 A (KRAFT EUROP R & D INC) 22 November 1990 (1990-11-22) claims; examples ---	1-3,5-7, 10
A	EP 0 540 085 A (UNILEVER PLC ;UNILEVER NV (NL)) 5 May 1993 (1993-05-05) claims ---	1
X	IDRIS NOR AINI ET AL.: "Chemical composition and physical properties of soft (tub) margarine sold in Malaysia. " JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY , vol. 73, no. 8, 1996, XP002231189 the whole document ---	13,14
X	EP 0 276 517 A (UNILEVER NV) 3 August 1988 (1988-08-03) page 3, line 43 - line 52 -----	13,14

## INTERNATIONAL SEARCH REPORT

Int      nal Application No

PCT/EP 02/12180

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 9704660	A	13-02-1997	AT 188844 T	15-02-2000
			AU 721705 B2	13-07-2000
			AU 6615796 A	26-02-1997
			CA 2227870 A1	13-02-1997
			DE 69606304 D1	24-02-2000
			DE 69606304 T2	10-08-2000
			DK 841856 T3	29-05-2000
			WO 9704660 A1	13-02-1997
			EP 0841856 A1	20-05-1998
			ES 2142598 T3	16-04-2000
			GR 3033046 T3	31-08-2000
			JP 11500320 T	12-01-1999
			JP 3083158 B2	04-09-2000
			PT 841856 T	30-06-2000
			US 5916608 A	29-06-1999
WO 9708956	A	13-03-1997	AT 207701 T	15-11-2001
			AU 723622 B2	31-08-2000
			AU 6821896 A	27-03-1997
			CA 2231787 A1	13-03-1997
			DE 69616572 D1	06-12-2001
			DE 69616572 T2	04-07-2002
			DK 848590 T3	27-12-2001
			WO 9708956 A1	13-03-1997
			EP 0848590 A1	24-06-1998
			JP 11511978 T	19-10-1999
			US 5916608 A	29-06-1999
			ZA 9607222 A	26-02-1998
EP 0864255	A	16-09-1998	GB 2323092 A	16-09-1998
			EP 0864255 A2	16-09-1998
WO 9603888	A	15-02-1996	AU 695202 B2	06-08-1998
			AU 3222695 A	04-03-1996
			CA 2196582 A1	15-02-1996
			DE 69505395 D1	19-11-1998
			DE 69505395 T2	29-04-1999
			DK 773722 T3	23-06-1999
			WO 9603888 A1	15-02-1996
			EP 0773722 A1	21-05-1997
			US 6071548 A	06-06-2000
DE 3907676	A	22-11-1990	DE 3907676 A1	22-11-1990
			GB 2229077 A , B	19-09-1990
EP 0540085	A	05-05-1993	EP 0540085 A1	05-05-1993
			AT 120340 T	15-04-1995
			AU 664567 B2	23-11-1995
			AU 2742492 A	06-05-1993
			CA 2081856 A1	01-05-1993
			DE 69201853 D1	04-05-1995
			DE 69201853 T2	31-08-1995
			JP 2621008 B2	18-06-1997
			JP 7213245 A	15-08-1995
			ZA 9208407 A	02-05-1994
EP 0276517	A	03-08-1988	AT 63043 T	15-05-1991
			AU 604294 B2	13-12-1990

Internal Application No  
PCT/EP 02/12180

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0276517	A	AU 1155788 A	15-07-1988
		CA 1340089 A1	20-10-1998
		DE 3769781 D1	06-06-1991
		WO 8804525 A1	30-06-1988
		EP 0276517 A1	03-08-1988
		EP 0329712 A1	30-08-1989
		JP 7083676 B	13-09-1995
		JP 1501916 T	06-07-1989